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By J. V. Robinson and W. W. Woods

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## SUMMARY

Mixtures of pure liquid hydrocarbons are capable of foaming. Nine hydrocarbons were mixed in pairs, in all possible combinations, and four proportions of each combination. These mixtures were sealed in glass tubes, and the foaming was tested by shaking. Mixtures of aliphatic with other aliphatic hydrocarbons, or of alkyl benzenes with other alkyl benzenes, did not foam. Mixtures of aliphatic hydrocarbons with alkyl benzenes did foam. The proportions of the mixtures greatly affected the foaming, the maximum foaming of 12 of 20 pairs being at the composition 20 percent aliphatic hydrocarbon, 80 percent alkyl benzene. Six seconds was the maximum foam lifetime of any of these mixtures.

Aeroshell 120 lubricating oil was fractionated into 52 fractions and a residue by extraction with acetone in a fractionating extractor. The index of refraction, foam lifetime, color, and viscosity of these fractions were measured. Low viscosity and high index fractions were extracted first. The viscosity of the fractions extracted rose and the index decreased as fractionation proceeded. Foam lifetimes and color were lowest in the middle fractions. Significance is attached to the observation that none of the foam lifetimes of the fractions or residue is as high as the foam lifetime of the original Aeroshell, indicating that the foaming is not due to a particular foaming constituent, but rather to the entire mixture.

## INTRODUCTION

A foam can be formed on aeronautical lubricating oils by bubbling in air, by shaking and beating, and by other mechanical processes. Such a foam has a lifetime varying from about  $\frac{1}{2}$  hour at room temperature to about 3 minutes at 100° C. The foaming behavior of lubricating oils is in marked contrast to the behavior of pure liquids, such as glycerol, diethylene glycol, or dibutyl phthalate, which will not form a column of foam under any conditions, even though of comparable viscosity. This is in accordance with the axiom that "pure liquids do not foam" (reference 1, p. 8).

By analogy with the foam stabilization of aqueous solutions by soaps and other surface-active materials, it has frequently been assumed that foams of lubricating oils are similarly stabilized by soaplike, or other surface-active materials (as discussed in the theory of foaming, reference 1). It is known from experience in this laboratory that certain additives in the oil may greatly increase the foam stability and foam volume, and furthermore that these additives concentrate in the foam and may be partially segregated by frothing off (p. 106, reference 1).

However, attempts made to froth off a surface-active agent from an undoped oil have been unsuccessful (p. 106, reference 1). The question has therefore been raised as to whether the hydrocarbon components of the oil could not be solely responsible for its foam-forming characteristics. In support of this contention, the experiments of Foulk and Barkeley (reference 2) should be cited on the film-forming tendencies of mixtures of water with benzene, nitrobenzene, ether, acetone, and methanol, as contrasted to the lack of film-forming tendency of the carefully purified liquids.

To test further the postulate that mixtures of pure hydrocarbons may foam, two series of experiments were performed. In the first, hydrocarbons of the best purity obtainable were mixed together and the foaming was tested. In the second, an undoped lubricating oil, Aeroshell 120, was fractionated by acetone extraction, under conditions not conducive to chemical changes, and the respective fractions were tested for foaming. It would be supposed that if some particular components were responsible for the foaming of Aeroshell 120, they would be concentrated in certain of the fractions, which should foam excessively. On the other hand, if the foaming were a property only of the mixture, each of the fractions should foam less than the original oil.

This work was conducted at Stanford University under the sponsorship and with the financial assistance of the National Advisory Committee for Aeronautics.

#### APPARATUS FOR ACETONE EXTRACTION

The acetone extractor was adapted from the design of Mair and Schicktanz (reference 3). The differences in the adaptation and the original design are principally in the relative sizes of certain parts (see fig. 1). The capacity of the lower reservoir, holding the oil sample, is 500 milliliters, and the acetone capacity above the oil is approximately 800 milliliters. The fractionating column is only 26 inches high whereas the original model had a column

four stories high. Also, instead of using a straight tube for the column, a tube with eight bulbs, as in a bulb condenser, is used. A small heater is used at the bottom of the convection stirring tube, to make stirring more positive and rapid. The entire apparatus is made of Pyrex glass.

#### PROCEDURE FOR ACETONE EXTRACTION

Five hundred grams of Aeroshell 120 was poured into the oil reservoir, through the condenser side of the apparatus. Acetone was put in until the level stood about halfway up the distilling bulb. The electric heater was turned on, and acetone distilled from the distilling bulb, condensed, ran down the return arm of the apparatus, and bubbled up through the oil. Air was bubbled slowly through the acetone from a capillary in the distilling bulb to prevent bumping. The extraction was carried on until the acetone in the distilling bulb became turbid. At first this took about 4 hours, but finally took about 36 hours, as the acetone solubility of the residue decreased. When the acetone in the distilling bulb became turbid, the electric heater was turned low, and the oil droplets were allowed to settle from the warm acetone for several minutes. The upper layer of acetone was then drawn off. The first sample drawn contained 500 milliliters of acetone, but this was decreased to 300 milliliters for subsequent samples. The extractor was run 24 hours per day for a period of about 10 weeks.

The oil was freed of the acetone by distilling off and recovering most of the acetone, then placing the oil in a small flask immersed in a boiling-water bath, with a very slow stream of dry air blowing over the oil surface. The oil purity was followed by means of its index of refraction. All of the samples were finished by evaporation at 100° C and about 30 millimeters of pressure, to constant index of refraction.

#### SOURCES AND PURITY OF MATERIALS

The conclusiveness of the first series of experiments to be described depends upon the purity of the hydrocarbons used. Since fractionation of hydrocarbons is very difficult, and better done in commercial units than in the ordinary laboratory, the samples were used as received from the sources. All of the hydrocarbons used were clear, colorless, and of low viscosity.

A very simple criterion for purity was used. The sample was tested for foaming by shaking. Those that formed a foam of appreciable lifetime were not used. The hydrocarbons used were the following:

Hydrocarbon	Source and grade
Aromatic	
Benzene	Merck's "Reagent" thiophene free
Toluene	Baker's CP Analyzed lot no. 12044
Xylene	Merck's "Reagent" grade
Cumene	Skelly Petroleum Company
Butyl benzene	Eastman Kodak Company
Aliphatic	
Isopentane	Phillips Petroleum Company, "pure grade 99 percent minimum"
Octene	Connecticut Hard Rubber Company
Octane	Connecticut Hard Rubber Company
Decane	Connecticut Hard Rubber Company

#### TEST PROCEDURE

The foam-testing method used was simply to shake the hydrocarbon mixture contained in a sealed glass tube, at room temperature, and to time the interval from cessation of shaking until all but two bubbles had disappeared. The last two bubbles were erratic in their behavior, and much better reproducibility was obtained by not counting them.

To prepare the sealed tubes, 15- by 1.8-centimeter test tubes were drawn out to form a narrow neck, the hydrocarbons were injected through the constriction from a syringe pipette, and the neck was sealed off. The hydrocarbon pairs were mixed in the proportions of 1:4, 2:3, 3:2, and 4:1, by measuring each hydrocarbon out from the syringe pipette, the total volume of the mixture being 10 cubic centimeters. The liquid did not touch the constriction, so no burning

took place when the tubes were sealed. Many of the tubes were prepared in duplicate, especially if the foaming was large, to eliminate the possibility of accidental contamination.

In determining the foam lifetimes, a standardized technique was used for shaking the tubes by hand. There is a personal factor here, but the reproducibility for one operator was good. One technician consistently obtained foam lifetimes about 30 percent greater than those reported here. The reported figures were all obtained by one technician and are the averages of 10 or more determinations in each case.

Because of the very small volume of some of the fractions obtained by acetone extraction of Aeroshell 120, the procedure used to test the foaming of the mixtures of hydrocarbons had to be modified. The fractions from Aeroshell were sealed in very short tubes, of 1.8-centimeter diameter, each tube containing 2 milliliters of oil, measured into the tube with a syringe pipette in the same manner as for the hydrocarbon mixtures. Because of the high viscosity of the oil fractions, no foam could be shaken at room temperature. The tubes were therefore immersed in a boiling-water bath, until they acquired the temperature of the bath. They were then removed and shaken quickly by hand in a standard manner, and again replaced in the boiling-water bath while the escape of the air was timed. The foam formed consisted of three to five layers of tiny air bubbles. The foam lifetime of each fraction was measured at least three times.

The indices of refraction were measured with an Abbe refractometer, at 20.0° C.

The viscosity was measured in an Ostwald-type capillary viscosimeter, at 25.00° C. The viscosimeter was especially constructed to require only 2 milliliters of oil. It was calibrated against a National Bureau of Standards viscosity reference oil of a viscosity of 10.06 stokes at 25.00° C; 2 milliliters of this oil had an efflux time of 142.5 seconds.

The color intensity was measured in a "Lumetron" photoelectric colorimeter. One of the fractions (fraction 23), pale amber in color, was selected arbitrarily as a reference standard, and the colorimeter was adjusted to read 100-percent transmission for this sample. The colorimeter is extremely sensitive, and was used with no filter of any kind. The samples which gave below 5-percent transmission appeared very dark. The color intensity was computed as equal to 100 minus the percent transmission. Very careful observations of different thicknesses of samples of the various fractions lead to the conclusion that all were visually indistinguishable in color, and that the obvious differences were in the color intensity only.

## RESULTS

All possible combinations of the nine hydrocarbons in pairs were tested. It was found that the greatest foaming occurred when the hydrocarbons containing the benzene ring were mixed with the aliphatic hydrocarbons. The foaming results are tabulated in tables I to IV.

Cumene had the longest foam life of any of the pure hydrocarbons, the foam lasting 1.0 second. Xylene was next with a foam life of 0.8 second. Three pure hydrocarbons gave foam lives of 0.6 second; one, 0.5 second; one, 0.4 second; and two, 0.3 second. It seems significant that none of the mixtures of aliphatic hydrocarbons (table II) nor the mixtures of alkyl benzenes (table III) had foam lifetimes in excess of 1.1 seconds. The foam lifetimes of mixtures of aliphatic hydrocarbons and mixtures of alkyl benzenes were only slightly greater than the foam lifetimes of the pure hydrocarbons. By contrast, 51 of the 100 mixtures of aliphatic hydrocarbons with alkyl benzenes had foam lifetimes in excess of 1.0 second, and 20 of the mixtures had foam lifetimes of 2.0 seconds or greater.

The proportions in which the hydrocarbons were mixed greatly affected their foaming. The mixtures showing the greatest foaming were the alkyl benzenes containing 20 percent aliphatic hydrocarbons, in 12 of the 20 pairs of alkyl benzenes with aliphatic hydrocarbons.

The results of the fractionation of Aeroshell 120 are shown in figure 2, in which the values given in table V are plotted. Foam lifetime, index of refraction, color intensity, and viscosity are shown. Viscosities were measured for 11 of the fractions. No attempt was made in figure 2 to indicate the sizes of the fractions. The weight of each fraction is listed in table V.

Figure 2 shows a high index of refraction for the first fractions, which drops sharply at fractions 7, 8, and 9, and then declines gradually to fraction 52. The foam lifetimes drop sharply from that of the original Aeroshell, and gradually rise again to the residue. There are several fractions that have high foam lifetimes compared with the general level of foaming of the fractions, but none that has as high a foam lifetime as the original Aeroshell. The color intensity shows the greatest fluctuation, the general tendency being that the lightest fractions are the intermediate ones.

## DISCUSSION

The question of whether mixtures of pure hydrocarbons can foam has both theoretical and practical significance. If foaming were

caused solely by polar-nonpolar stabilizing agents in the lubricating oil, they could possibly be removed by some process. If, however, the foaming is a property of the mixture of hydrocarbons, an oil of petroleum source can hardly be made nonfoaming by refining. Only antifoaming additives can chemically defoam the oil in this case.

The principal question therefore is whether the experimental work described, in which mixtures of hydrocarbons are shown to foam, is definitive. This in turn hinges on whether the hydrocarbons may be regarded as devoid of a "foaming agent." Most of the hydrocarbons used cannot be regarded as a single chemical species, the measurable foam lifetimes of most of them being a priori proof of this. Furthermore, mixtures of pairs of similar hydrocarbons (i.e., aliphatic with aliphatic, and alkyl benzenes with alkyl benzenes) foam at most only slightly more than the separate samples.

All current foam theories require that the composition of the liquid film surface be different from the composition of the bulk liquid, and that some structure be present at the film surface to delay the flow of liquid from the film or to prevent the instantaneous rupture of the films, or both. Suppose that several of the hydrocarbons used in these experiments contained traces of soaplike or resinous substances which might stabilize foams. To fit the experimental results, these substances must not adsorb on the surface of unmixed hydrocarbons, or on the surface of mixtures of like hydrocarbons, but must adsorb on the surface of mixtures of unlike hydrocarbons. However, the mechanism of the latter process may be visualized, by analogy with solutions from which a solute may be precipitated by adding a solvent of lesser dissolving power. It is harder to explain the failure to adsorb of the hypothetical soaplike or resinous material at the film surfaces of the unmixed hydrocarbon.

A "foam lifetime" of 0.3 second, as in the cases of pure toluene and of pure octane represents a minimal foaming tendency; using the described method of measurement, the foaming of pure water is as great. The mixture that foamed the most, of these two hydrocarbons, however, has a foam lifetime of 1.9 seconds. That this effect is not illusory may be seen by holding the six sealed tubes in one hand, with pure toluene at one end and pure octane at the other, and the mixtures, in steps of 20 percent, in between. The tubes are then shaken simultaneously. The almost immediate disappearance of bubbles in the end tubes, contrasted to the graduated times of disappearance of bubbles in the intermediate tubes, is most striking, even though the maximum time for bubble disappearance is only about 2 seconds.

The most persuasive argument against a fortuitous contamination of the hydrocarbons by nonhydrocarbon substances responsible for the foaming behavior is in the regularity of the concentration of the



mixtures of alkyl benzenes with aliphatic hydrocarbons at which maximum foaming occurs. The mixtures of octene, octane, and decane with benzene, toluene, xylene, cumene, and butyl benzene (15 combinations) show the maximum foaming at a composition of 20 percent by volume of the aliphatic hydrocarbon in 11 of the 15 cases. Three of the four exceptions are with octene, an unsaturated hydrocarbon. Such regularity, if the foaming is ascribed to the effect of impurity, requires an amazing homogeneity in the properties of the impurities found in these hydrocarbons of widely varying source.

It is the authors' opinion that the foaming is a property of the mixture of hydrocarbons, and is not the result of impurities. It is suggested that there is an adsorption of one of the hydrocarbons at the surface, and that molecules which may be close-packed at the surface favor foaming. Mixtures of similar hydrocarbons would have little tendency to "squeeze out" any component, all being essentially equivalent in their cohesion. Mixtures of aliphatic and aromatic or condensed ring molecules, however, would have a tendency to force one species of molecule to the surface, there being a difference in cohesion. An unsuccessful attempt was made to correlate foaming with the internal pressures of the hydrocarbons, calculated from their van der Waals' constants, specific volumes, and molecular weights. The importance of the steric configuration of the molecules for such surface packing is apparent. The proportion of the mixture showing the greatest foaming tendency would be that at which the adsorption of the species capable of the closest packing was greatest.

A few observations were made in the course of these experiments which suggested that strongly foaming mixtures of hydrocarbons may be formulated. Solutions of naphthalene (avoided because of the lack of a simple test for its purity) in hydrocarbons such as toluene will foam. It is suspected that solutions of naphthalene in hydrocarbons such as octadecane, containing toluene, cumene, or butyl benzene to increase the solubility of the naphthalene, might have properties of viscosity and foaming similar to lubricating oils. Compounds such as methyl naphthalenes would be even better than naphthalene, if available sufficiently pure. Samples of methyl naphthalene obtained were strongly foaming in themselves.

Because of the considerable fluctuations in measurement caused by the necessity of using small samples, it is difficult to make a quantitative estimate of the results obtained by the acetone extraction of Aeroshell. There can be no doubt, however, that there is no foaming agent even approximately comparable in effectiveness with that found in certain oils containing lubricating additives. Furthermore, the failure to foam excessively of any of the oil fractions obtained is believed to indicate that there is no component in the oil specifically responsible for the foaming.

Fractions 1, 2, 21, and 23 have about the same foam stability as Aeroshell, taking into account their lower viscosities. All of the other fractions have about half of the foam stability of Aeroshell. Aside from the large fluctuations of the foam stability of alternate fractions, there is a gradual downward trend to the foam lifetime over viscosity, terminating with a very low value for the residue.

Mair and Schicktanz (reference 3) found that the aromatic constituents of lubricating oil are the most soluble in acetone. The gradual slope of the foam-stability curve would indicate that, as the concentration of aromatic hydrocarbons decreased, the foam stability decreased.

The change of color of the fractions suggests that the distribution of color is very sensitive to changes in composition of the oil. The color tended to accumulate in the residue, indicating preferential solubility in paraffin rather than in aromatic hydrocarbons. Foaming evidently has nothing to do with color; the least-colored oils of these fractions foam the most. This conclusion is confirmed by the observations previously reported (reference 1) that the foam stability divided by the viscosity was about the same for white oils and lubricating oils.

#### CONCLUDING REMARKS

It is concluded that mixtures of pure hydrocarbons may have the property of foaming. The nature of the hydrocarbons and their proportions in the mixtures determine the degree of foaming.

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Stanford University, Calif., August 8, 1945

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2. Foulk, C. W., and Barkeley, John E.: Film Formation by Pure Liquids. Ind. and Eng. Chem. (Ind. Ed.), vol. 35, no. 9, Sept. 1943, pp. 1013-1016.
3. Mair, Beveridge J., and Schicktanz, Sylvester T.: Extraction, with Acetone, of Substantially Constant-Boiling Fractions of a "Water-White" Lubricating Oil. Res. Paper RP953, Jour. Res. Nat. Bur. of Standards, vol. 17, no. 6, Dec. 1936, pp. 909-922.

TABLE I.- FOAMING OF PURE HYDROCARBONS

Hydrocarbon	Time for foam to collapse to last pair of bubbles (sec)
Aliphatic hydrocarbon	
Isopentane	0.4
Octene	.6
Octane	.3
Decane	.6
Alkyl benzene	
Benzene	0.6
Toluene	.3
Xylene	.8
Cumene	1.0
Butyl benzene	.5



TABLE II.- FOAMING OF MIXTURES OF ALIPHATIC HYDROCARBONS

Aliphatic hydrocarbon	Percent isopentane				Percent octane				Percent octene				Percent decane			
	20	40	60	80	20	40	60	80	20	40	60	80	20	40	60	80
	Time for foam to collapse to last pair of bubbles (sec)															
Isopentane	---	---	---	---	0.8	0.7	0.6	0.5	0.8	0.6	0.4	0.4	1.1	0.9	---	0.6
Octane	0.5	0.6	0.7	0.8	---	---	---	---	.6	.6	.6	.6	.7	.7	0.6	.7
Octene	.4	.4	.6	.8	.6	.6	.6	.6	---	---	---	---	.6	.6	.5	.7
Decane	.6	---	.9	1.1	.7	.6	.7	.7	.7	.5	.6	.6	---	---	---	---



TABLE III.- FOAMING OF MIXTURES OF ALKYL BENZENES

Alkyl benzene	Percent benzene				Percent toluene				Percent xylene				Percent cumene				Percent butyl benzene			
	20	40	60	80	20	40	60	80	20	40	60	80	20	40	60	80	20	40	60	80
	Time for foam to collapse to last pair of bubbles (sec)																			
Benzene	---	---	---	---	0.7	0.7	0.7	0.7	0.8	0.8	0.9	1.0	0.8	0.9	1.0	1.0	0.8	0.7	0.7	0.7
Toluene	0.7	0.7	0.7	0.7	---	---	---	---	.7	.9	.9	1.0	.8	.9	.9	.9	.6	1.0	.9	.6
Xylene	1.0	.9	.8	.8	1.0	.9	.9	.7	---	---	---	---	1.0	1.0	1.0	.9	.8	.8	.7	.8
Cumene	1.0	1.0	.9	.8	.9	.9	.9	.8	.9	1.0	1.0	1.0	---	---	---	---	.9	1.0	.8	.8
Butyl benzene	.7	.7	.7	.8	.6	.9	1.0	.6	.8	.7	.8	.8	.8	.8	1.0	.9	---	---	---	---



TABLE IV.- FOAMING OF MIXTURES OF ALIPHATIC HYDROCARBONS WITH ALKYL BENZENES

Alkyl benzene	Percent isopentane				Percent octene				Percent octane				Percent decane			
	20	40	60	80	20	40	60	80	20	40	60	80	20	40	60	80
	Time for foam to collapse to last pair of bubbles (sec)															
Benzene	0.7	0.8	1.0	0.8	1.7	1.3	1.0	0.8	1.9	1.6	1.1	0.8	3.8	2.1	1.0	0.7
Toluene	.7	.6	.6	.6	1.4	1.6	1.1	.9	1.9	1.8	.8	1.0	4.0	2.4	1.3	.7
Xylene	.9	.9	1.0	1.1	1.6	1.4	1.4	1.0	1.8	1.8	1.3	1.0	3.4	2.5	1.5	.8
Cumene	.7	.8	1.0	1.3	1.4	1.5	1.3	1.0	2.0	2.0	1.8	1.1	3.2	3.0	2.1	1.2
Butyl benzene	.9	1.3	1.4	1.8	1.7	2.3	2.2	1.9	1.9	2.9	2.6	2.0	6.1	6.0	4.6	2.6


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TABLE V.- FRACTIONATION OF AEROSHELL 120 BY ACETONE EXTRACTION

Fraction	Weight (g)	Index of refraction at 20.0° C	Foam life (sec)	Viscosity at 25° C (stoke)	Color intensity
1	30.9	1.5010	140	----	45
2	11.3	1.4990	140	----	62
3	28.7	1.4993	65	3.66	25
4	24.4	1.4928	100	----	1
5	----	1.5008	45	----	--
6	----	1.4990	80	----	81
7	----	1.4930	---	----	98
8	----	1.4924	90	----	37
9	----	1.4898	90	----	12
10	4.8	1.4992	80	----	24
11	3.7	1.4900	90	3.81	24
12	4.0	1.4900	90	----	33
13	3.9	1.4888	100	----	3
14	3.8	1.4897	70	----	7
15	2.0	1.4887	120	----	12
16	.3	1.4887	60	----	38
17	3.0	1.4884	100	4.21	20
18	4.4	1.4883	70	----	44
19	6.0	1.4881	70	4.64	37
20	8.7	1.4880	70	----	6



TABLE V.- FRACTIONATION OF AEROSHELL 120 BY ACETONE

## EXTRACTION - Continued

Fraction	Weight (g)	Index of refraction at 20.0° C	Foam life (sec)	Viscosity at 25° C (stoke)	Color intensity
21	10.3	1.4872	210	4.65	3
22	9.1	1.4872	140	----	28
23	10.6	1.4878	240 → 100	----	6
24	12.9	1.4868	100	4.50	-3
25	10.3	1.4873	140 → 70	----	-2
26	12.1	1.4870	80	----	11
27	8.2	1.4870	70	4.97	16
28	8.3	1.4867	80	----	-1
29	7.5	1.4870	160 → 70	----	1
30	6.7	1.4870	100	----	8
31	6.9	1.4869	110	----	-5
32	6.7	1.4869	80	----	24
33	7.7	1.4868	180 → 60	----	26
34	6.2	1.4873	140 → 80	----	17
35	6.7	1.4872	120	----	52
36	4.9	1.4872	80	----	80
37	3.2	1.4864	100	----	92
38	5.8	1.4866	120	6.63	83
39	3.8	1.4872	110	----	84
40	4.5	1.4869	150	----	67

TABLE V.- FRACTIONATION OF AEROSHELL 120 BY ACETONE

EXTRACTION - Concluded

Fraction	Weight (g)	Index of refraction at 20.0° C	Foam life (sec)	Viscosity at 25° C (stoke)	Color intensity
41	4.8	1.4862	260 → 160	6.61	92
42	3.5	1.4861	180 → 120	-----	86
43	4.6	1.4867	120	-----	84
44	3.2	1.4867	70	-----	95
45	3.6	1.4869	120	-----	89
46	3.6	1.4870	430 → 180	9.50	97
47	3.5	1.4869	170 → 90	-----	84
48	3.5	1.4866	180	-----	63
49	3.2	1.4872	170 → 90	-----	69
50	3.3	1.4872	200 → 130	9.13	65
51	3.5	1.4872	100	-----	76
52	3.9	1.4872	330 → 210	-----	95
Residue	147.1	-----	200	37.0	93
Aeroshell	-----	1.4907	335	9.00	50



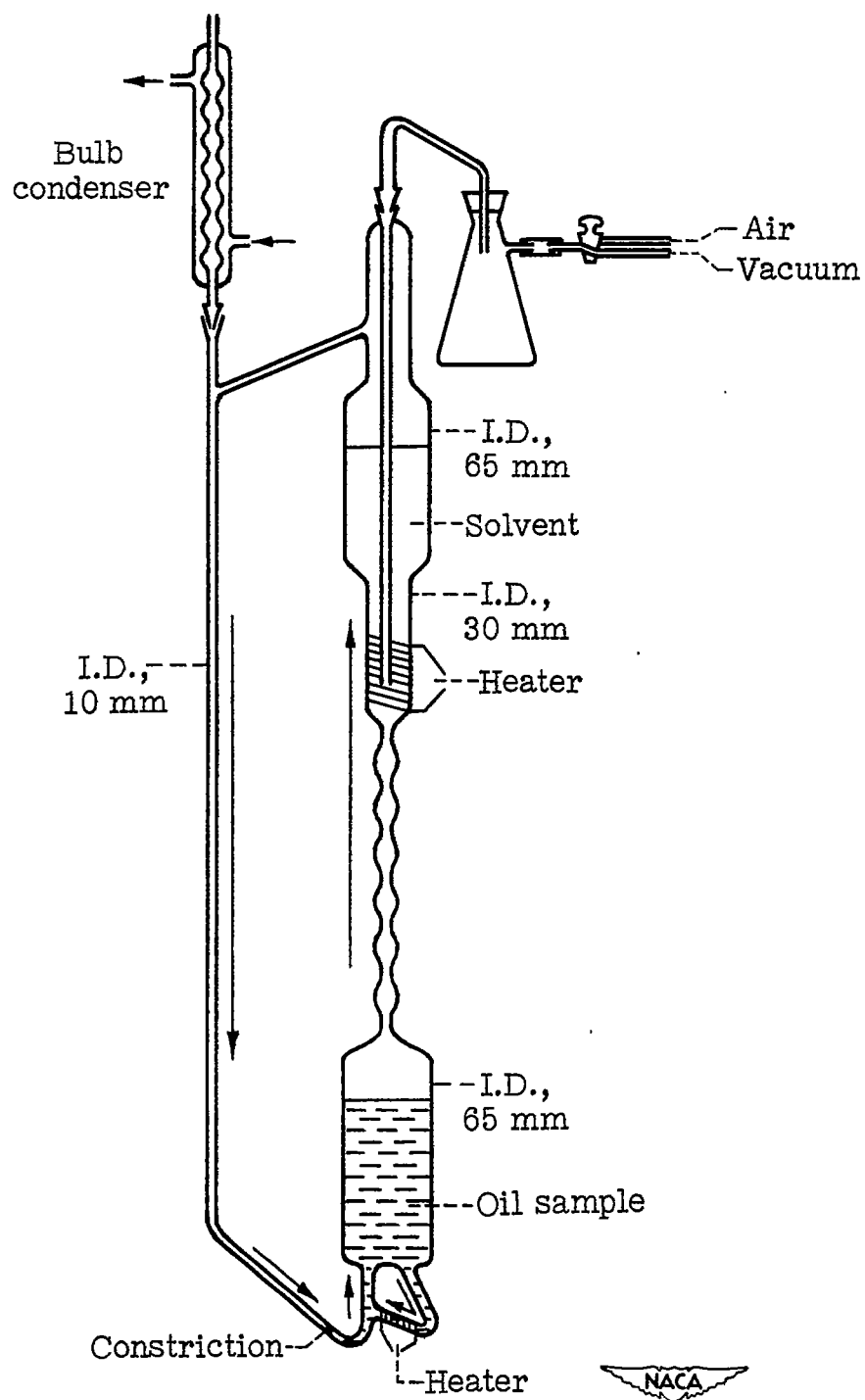


Figure 1.- Liquid extractor for fractionation of oils.

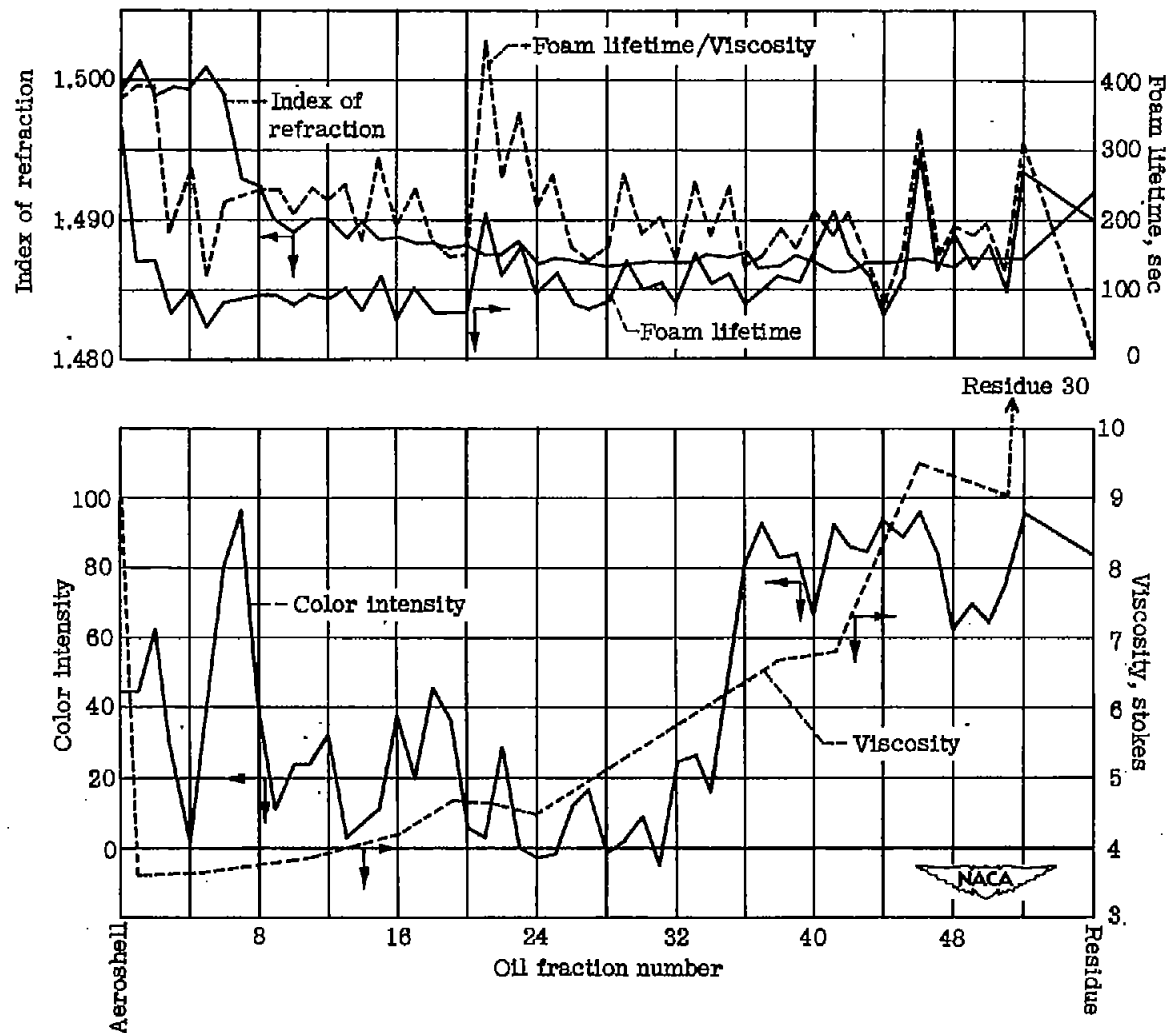


Figure 2.- Aeroshell 120 fractionated by acetone extraction.